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Hardware Article

Methods to simplify cooling of liquid Helium cryostats

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ABSTRACT

Liquid Helium is used widely, from hospitals to characterization of materials at low temperatures. Many experiments at low temperatures require liquid Helium, particularly when vibration isolation precludes the use of cryocoolers and when one needs to cool heavy equipment such as superconducting coils. Here we describe methods to simplify the operations required to use liquid Helium by eliminating the use of high pressure bottles, avoiding blockage and improving heating and cooling rates. First we show a simple and very low cost method to transfer liquid Helium from a transport container into a cryostat that uses a manual pump having pumping and pressurizing ports, giving a liquid Helium transfer rate of about 100 liters an hour. Second, we describe a closed cycle circuit of Helium gas cooled in an external liquid nitrogen bath that allows precooling a cryogenic experiment without inserting liquid nitrogen into the cryostat, eliminating problems associated to the presence of nitrogen around superconducting magnets. And third, we show a sliding seal assembly and an inner vacuum chamber design that allows inserting large experiments into liquid Helium.

Specifications tables:

Hardware name	Helium transfer system based on manual pump
Subject area	• Materials Science • Physical property measurements • Cryogenics of magnetic resonance equipment
Hardware type	• Device to transfer liquid Helium • Maintenance and preparation of systems for physical property measurements at low temperatures
Open source license	CERN OHL
Cost of hardware	30 $
Source file repository	https://osf.io/e6k7r/

(*continued on next page*)

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2468-0672/

Hardware name	Closed cycle Helium gas precooling system
Subject area	• Materials Science • Physical property measurements
Hardware type	• Precooling equipment to liquid nitrogen temperatures • Physical property measurements at low temperatures
Open source license	CERN OHL
Cost of hardware	1500 $
Source file repository	https://osf.io/e6k7r/

Hardware name	Sliding seal and inner vacuum chamber for rapid sample exchange
Subject area	• Materials Science • Physical property measurements
Hardware type	• Precooling equipment to liquid nitrogen temperatures • Physical property measurements at low temperatures
Open source license	*CERN OHL*
Cost of hardware	*200* $ Without taking into account machining costs, which might be significant for this device in some countries.
Source file repository	https://osf.io/e6k7r/

1. Hardware in context

Much of the innovative work in cryogenics is now devoted into dry or closed-cycle cryogenic systems, driven by the development of thermoacoustic systems and the improvement of thermodynamic machines [1]. However, the improvements have not displaced liquid Helium, but considerably facilitated its development and use in different fields. For example, magnetic resonance imaging cryostats include coolers providing improved shielding that nearly zero the consumption of liquid Helium[2,3]. In research laboratories, large systems including superconducting magnets require most often liquid Helium, because cooling using the huge enthalphy of Helium gas is unmatched with respect to any other cooling method. In addition, there is an increasing need for experiments in which the mechanical vibrations are held to a minimum, for which the use of liquid Helium is unavoidable [4–11]. Therefore, research in wet cryogenics is needed to improve the performance of systems using liquid Helium.

In particular, cooling of large cryostats bears some inherent problems related to the manipulation of liquid Helium. There are many laboratory books and notes that are there to help the users, in addition to usual manuals provided by companies delivering cryostats [12,13]. Most of these manuals explain transfer of liquid Helium and sample turn-around methods. But several problems are repeatedly mentioned. These are about handling of liquid Helium bottles and transfer methods, inserting and cooling to liquid Helium temperatures large inserts, often containing a dilution refrigerator unit, and precooling to liquid nitrogen temperatures. For example, as we explain below, tubes can be blocked when liquid nitrogen has been used and not fully removed before transferring liquid Helium, or Helium leaks appear when inserting large systems into liquid Helium.

Here we discuss possible solutions to these issues, which we developed in the use of a set of five large dilution refrigerator units that are equipped with Scanning Tunneling Microscopes. The microscopes have been partially described in detail in Ref. [14]. A cryogenic set-up with a three axis vector magnet and associated electronics for the magnet and the microscope has been described in Ref. [15]. The solutions described here have been developed more recently. The Helium transfer manual pump has not been reported yet, to our knowledge. The liquid nitrogen precooling device has been reported in Ref. [16] in a realization that includes a specific compressor and heat exchanging system. Here we show a much simpler realization, using in particular a compressor that can be found in common stores. The improvements in sliding seal assembly and vacuum chamber have not been reported to our knowledge.

2. Hardware description

Potential advantages of using the described hardware:

- Eliminating high pressure Helium equipment from cryogenic laboratories.
- Avoiding blockage and problems with transfer of liquid nitrogen into cryostats.
- Improving sample turn around time in cryogenic devices.

2.1. Helium transfer system based on manual pump

Liquid Helium is usually transferred from a transport Dewar to a cryostat using a transfer tube and applying an overpressure to the transport Dewar. An acceptable flow rate is usually of about 50 liters of liquid Helium for half an hour, which makes a volumetric flow rate $Q \approx 30$ ml/s. With a cross section of a pipe which typically is of about $d = 10$ mm^2, this gives a velocity of liquid Helium of a few m/s. The force needed to produce Helium flow is the force needed to overcome height differences in the liquid Helium levels of both containers and to overcome friction. An overpressure of 0.2–0.4 bar in a container of typically a quarter of a square meter cross section is enough to transfer liquid Helium into the cryostat. Although the Reynolds number can be as high as 10^6, losses due to friction do not hamper significantly the transfer process[17]. The high Reynolds number makes liquid Helium flow a rather unusual process, which is however routinely carried out in laboratories.

To produce the overpressure of, say, 0.5 bar, one can simply introduce Helium gas into the transport Dewar by some means. A few simple calculations show that the evaporation produced in the liquid by the arrival of a small amount of hot gas is by far enough to produce an overpressure in the transport Dewar. For example, in a usual 100 liters transport Dewar, there is somewhat less than a m^3 room for the gas. The latent heat of vaporization of liquid Helium is of about 21 kJ/kg. The enthalpy of Helium gas between room temperature and liquid Helium temperatures is of about 1.5 MJ/kg. Cooling of a kg of gas to liquid Helium temperatures requires evaporating about 75 kg of liquid. Taking into account the different densities, this implies that to achieve the required overpressure, one just needs to evaporate about a liter of liquid and that this can be done by cooling from ambient to liquid Helium temperature just about 12 liters of Helium gas. Therefore, one needs to insert into the transport Dewar a small amount of Helium gas at room temperature to produce the required overpressure.

A usual solution is a small laboratory membrane compressor, which allows in principle for automated operation, but it is often not very practical. The flow rate of a small device is of about 10 liters per minute or less. This implies that the gas has enough time to thermalize inside the transport Dewar and remain at the upper level, without really evaporating the liquid. Furthermore, care has to be taken to make sure that Helium is not pumped out of the recovery line when it is not needed, which would imply losses of Helium gas. In all, the pumping speed of small membrane compressors makes that the needed overpressure is obtained in several minutes. Furthermore, the compressor is often stuck by impurities or debris from the recovery line. If the experiment can continue being operated during the liquid Helium transfer, a membrane compressor can be a good solution, because there is no need to transfer at a high rate. However, vibration sensitive experiments usually cannot be made during Helium transfer. There are other constraints, such as multiple users of the same liquid Helium transport container or avoiding overpressure in the recovery line when users are transferring at the same time. Therefore, often a fast transfer is desired. The solution is to use Helium gas bottles pressurized to 200 bar with a pressure reducing station with a manometer. However, high pressure gas bottles requires safety management and it is quite easy to loose large amounts of gas by bad manipulation.

Rubber bladders can be used when slow filling rates are required[13]. The bladder is inflated with the small overpressure of the Helium transport container. By pushing on the bladder, a few liters of Helium gas are moved inside the transport container and the obtained flow of gas evaporates a small amount of liquid, which leads to an increase in pressure. Small pressures can be nicely regulated this way, but when high transfer rates are needed, the bladder becomes too big to operate.

Recently, manual gas pumps with an entry and an exit port have been made available in the market. These are used to fill and empty large containers with air, such as boats or camping appliances. They pump gas volumes between 5 and 10 liters per stroke and a maximal pressure difference of order of 0.5 bar (see for examplehttps://www.decathlon.co.uk/52-l-hand-pump-id_8243066.html). We have adapted one of such pumps to collect Helium from the recovery of the cryostat and insert the gas into the transfer Dewar. The pump has the speed and size to move enough Helium gas inside the transport Dewar and produce the required liquid Helium evaporation. We show schematically in Fig. 1 the whole arrangement. A few strokes to the pump are enough to produce the required increase in the pressure of the transport Dewar and fill a cryostat to with a rate of about 100 liters an hour.

The changes that have to be made at the pump are minimal and consist of gluing connections and pressure gauges, exchanging the grease used to lubricate the mechanical parts of the pump by vacuum grease and making threads leak tight. We describe these changes in the instructions section.

2.2. Closed cycle Helium gas precooling system

As mentioned above, the latent heat of vaporatization of liquid Helium is small (21 kJ/kg), so that precooling the system to liquid nitrogen allows reducing considerably the Helium boil-off. On the other hand, the melting point of liquid nitrogen is just at 63 K, quite close to the temperature of liquid nitrogen at ambient pressure, 77 K, and the latent heat of fusion is of about 26 kJ/kg. This implies that, if the liquid nitrogen is not fully removed when the system has cooled, a sizeable amount of liquid Helium is needed to solidify the liquid nitrogen and cool down to liquid Helium temperatures. For example, if just an amount of liquid nitrogen of order of a cup, or of about 100 g of liquid nitrogen, is left in the system, 2.6 kJ are needed to solidify it, which implies evaporating about a kg, or about 10 l, of liquid Helium.

Removing all liquid nitrogen is not easy. One can use a tube inserted down to the bottom of the cryostat and an overpressure, so that nitrogen is transferred back outside the cryostat. However, the tube must be designed in such a way as to reach the bottom of the cryostat so that all nitrogen can be removed. In addition, the cryostat must be flushed and pumped several times [13]. There is always a risk of leaving small amounts of nitrogen in thin tubes, and this can lead to blockage when cooling to liquid Helium. Furthermore, nitrogen might get trapped inside superconducting magnets, which produces strain that might lead to quench during operation.

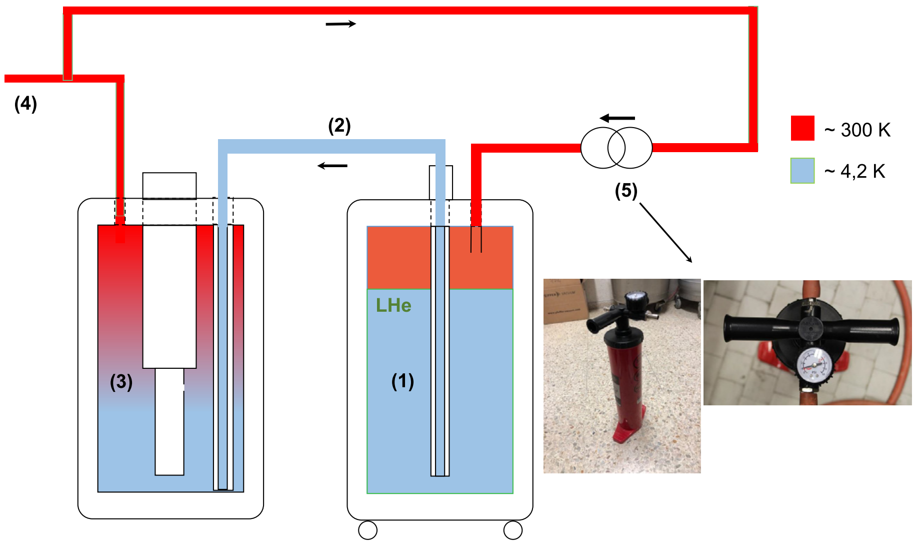

Fig. 1. Scheme of the liquid Helium transfer system. We show schematically a liquid Helium transport Dewar (1), a transfer tube (2) and a cryostat (3). The cryostat is connected to the Helium recovery line (4). The transport Dewar (1) is connected to the recovery line (4) through a manual pump (5). The manual pump (5) is shown as a photo in the lower right part of the figure. The manual gas pump has clear entry and exit ports. Connectors for rubber tubes are glued to the ports. About five strokes of the manual pump are needed to fastly send Helium gas into the Dewar. The Helium gas is cooled by the liquid Helium, which evaporates and increases the pressure in the transport Dewar. The evaporation produces a small overpressure of about 0.5 bar, with which liquid Helium is transferred to the cryostat. Using this pump, 100 liters can be transferred in about an hour.

To avoid these difficulties, we have designed a precooling system as schematically described in Fig. 2. Helium gas is circulated within a close circuit through a heat exchanger in liquid nitrogen, then inserted into the cryostat and flows again into the circuit after cooling the cryostat. We force Helium gas at room temperature through a serpentine immersed in liquid nitrogen and insert the outcoming cold gas using a usual Helium transfer tube into a cryostat with a superconducting coil inside. The warm Helium gas exiting the cryostat is again inserted into the circuit.

The same method has been proposed in the past[16], with, however, a rather complex heat exchanger and a multiple stage blade compressor.

To discuss the working principle of the system, let us consider an example with a system whose cooling would be equivalent to cool about 50 kg of copper. The heat of vaporization of liquid nitrogen is of about 200 kJ/kg. Density of liquid nitrogen is of about 800 kg/m^3, so the vaporization of a m^3 of liquid provides 160 MJ. Cooling 50 kg of copper from room temperature down to liquid nitrogen requires about 3 MJ (63 kJ per kg of copper), or 20 liters of liquid nitrogen.

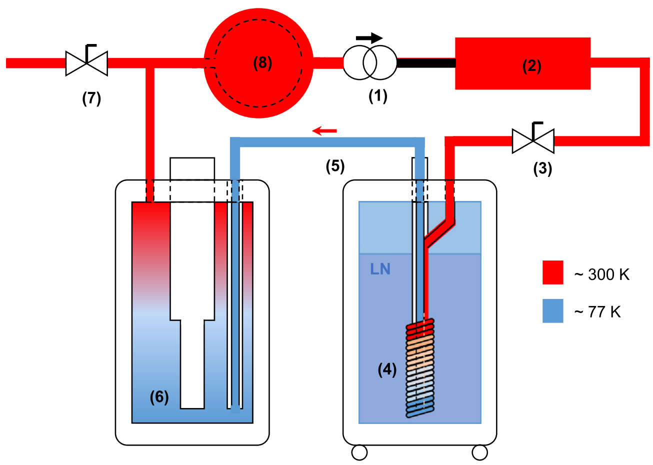

Fig. 2. Scheme of the precooling system. A compressor (1) is connected to a pressure container (2). A tube connects the container (2), through a regulating valve (3) to a heat exchanger immersed into liquid nitrogen (4). Helium flows out of the heat exchanger through a usual Helium transfer tube (5) into the cryostat (6). A valve (7) connects the recovery of the cryostat to the Helium recovery line of the laboratory. This valve is closed during operation and can be used to fill the whole circuit with Helium gas. The recovery of the cryostat is connected to a balloon (8) that reduces the time needed to operate the compressor (1) and allows for continuous flow.

On the other hand, Helium gas has a specific heat at constant volume of about 3 kJ/kgK. Taking a density of 0.17 kg/m^3, this gives 0.5 kJ/m^{3}K. Assuming that Helium follows the ideal gas law and the specific heat is constant with temperature, we estimate that cooling Helium gas from room temperature to liquid nitrogen requires removing 100 kJ per m^3 of gas. If we use liquid nitrogen to cool the gas, we need about 0.6 liters of liquid nitrogen to cool a m^3 of Helium gas to liquid nitrogen temperatures.

To cool the amount of copper mentioned above (50 kg) to liquid nitrogen, we need to evaporate at least 20 liters of liquid nitrogen. Here we do this by using Helium gas that has been previously thermalized in the liquid nitrogen. This implies that we need to circulate about 30 m^3 of Helium gas through liquid nitrogen. With a circulation of 10 m^3/h of Helium gas thermalized at liquid nitrogen temperatures, the required temperature drop is achieved within some hours. This circulation rate can be easily achieved using a small size commercial membrane compressor available in a usual store.

It is relatively easy to build a heat exchanger (Fig. 3(a)) that allows cooling such an amount of Helium gas with liquid nitrogen. A one-dimensional heat exchanger consisting of a 1 m long tube copper tube with a diameter of the order of a cm suffices to achieve the desired cooling. (See Fig. 4).

It is very useful to use an entry port that fits usual diameters of Helium tubes. In Fig. 3(a) we provide an adaptor making a very compact design. The design consists of a leak tight system of O-rings that is duplicated with two different internal diameters. The smaller diameter is simply inserted into the larger diameter in such a way that the two most usual transfer tube diameters (12 and 9 mm) can be used. We provide construction details in the designs accompanying this work.

The circuit requires an expansion Helium gas by the pressure difference which is established between the input in the cryostat and the exit. The Joule-Kelvin coefficient of Helium gas is small, of about 0.06 K/bar[18], so that the expansion implies a minimal heating (about 60 mK).

We have used this system to cool a cryostat with a superconducting coil and a dilution refrigerator and could reach temperatures of about 100 K overnight (see validation results in Fig. 5). After that, we could immediately start transferring Helium.

Of course the Helium gas should not be pumped from a large reservoir such as the recovery line, because an error in the operation might lead to large Helium losses. Instead, one needs to use a closed circle circulation, which requires some design. First it is important to realize that, if a simple commercial small sized compressor is operated all the time and without stop, it will burn down the motor. Therefore, the circuit is not a simple closed annular flow through a compressor and requires intermediate high and low pressure containers, described in Fig. 2. The pressure difference estab-

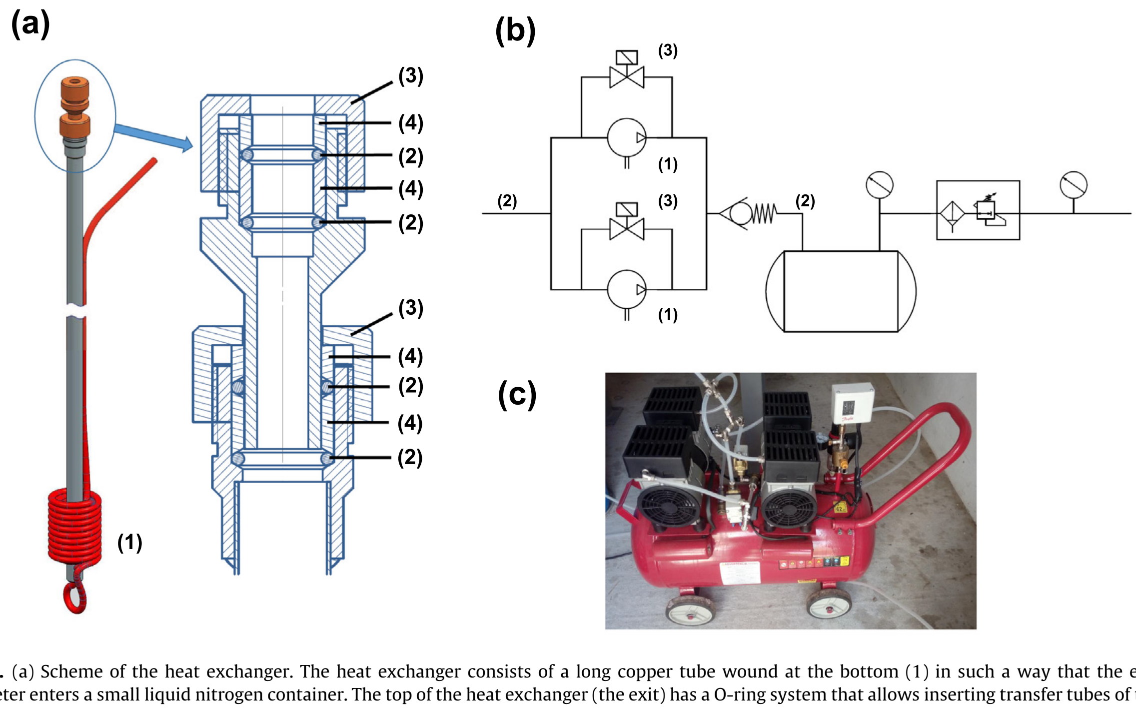

Fig. 3. (a) Scheme of the heat exchanger. The heat exchanger consists of a long copper tube wound at the bottom (1) in such a way that the external diameter enters a small liquid nitrogen container. The top of the heat exchanger (the exit) has a O-ring system that allows inserting transfer tubes of the two most common diameters (9 mm and 12 mm). It consists of four O-rings (2) that are held by threaded brass caps (3) and internal brass caps (4) that are machined at an angle at the ends to compress the O-ring to the transfer tube that is to be inserted. (b) Scheme of the compressor. The system (1) is a commercially available set of two membrane compressors mounted in parallel. The entry and exit ports (2) have been sealed using Araldite. The valves (3) have been exchanged by leak-tight vales and we have added a system that allows equilibrating pressures before re-starting the compressor. (c) Photography of the modified compressor with pressure container.

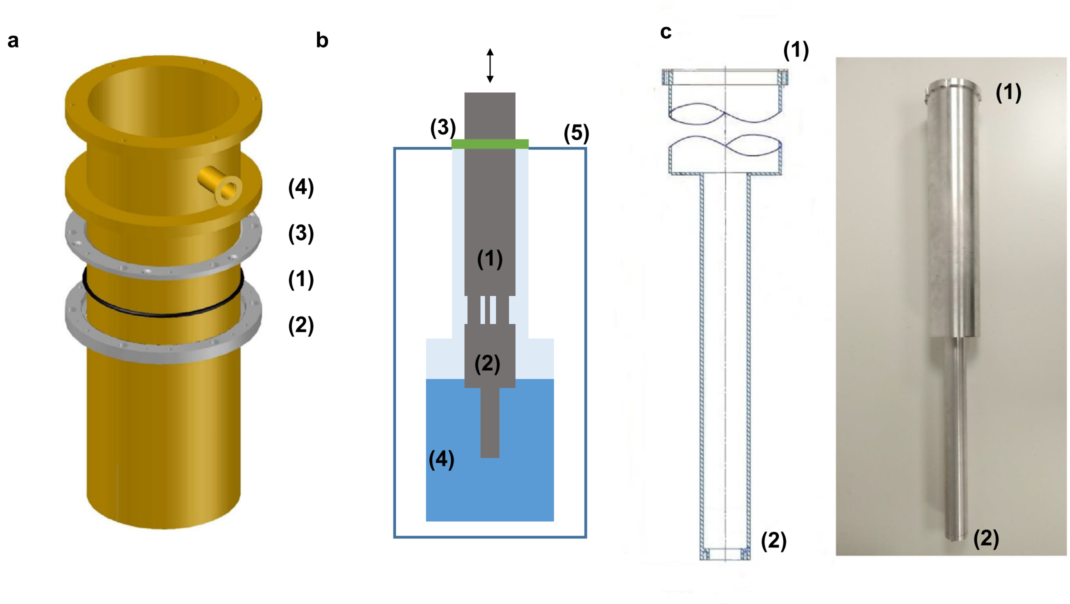

Fig. 4. (a) Schematics of the sliding seal assembly consisting of an O-ring (1) and a bottom stainless steel piece (2) and a cap (3). The bottom part has threaded holes that allow fixing (3) in such a way as to compress the O-ring and additional holes allowing to fix the insert into the cryostat. (b) Schematics of a sliding seal (1) and innver vacuum chamber (2) inserted through the O-ring assembly (3) into a liquid Helium bath (4) inside a cryostat (5). (c) Schematic drawing and photo of an Aluminium inner vacuum chamber, with the top (1) Indium seal assembly to connect the chamber to the cryostat and a bottom Indium seal (2) as a tap.

lished by the flow of Helium gas occurs at the transfer tube (Fig. 2) mostly. Using Hagen-Poiseuille, we obtain about 1 bar considering a transfer tube of 2 mm diameter and 3 m length. To avoid using the compressor all times, we use a pressurized container after the compressor ((2) at Fig. 2). We use a pressure guage and switch to start the compressor whenever the pressure decreases below 1.5 bar and to stop it when it reaches 4 bar at the pressurized container. A regulating valve at the exit of the pressurized container ((2) at Fig. 2) limits the flow. In order to have enough gas to establish the overpressure each time the compressor is switched on again, we require a Helium balloon on the low pressure side ((8) at Fig. 2). The size of the balloon and of the pressurized container determine the length in time that the compressor needs to be operated. With a circulation of, for example, 10 m^3/h, or 166 l/min, and a pressurized container of about 50 liters, a few hundred liters of gas flow out of the container each couple of minutes. The compressor operates then for a fraction of a minute each two minutes. We used a balloon of about 500 liters and obtained acceptable operating rates for the compressor. When the gas flows out of the pressurized container, it slowly fills the balloon and when the pressure of the container reaches 1.5 bar, the compressor takes the gas from the balloon and fills the pressurized container.

We used a simple commercial oil-free compressor with a flow rate of about 10 m^3/h. To achieve such a flow rate, the device consists of a couple of membrane compressors connected in parallel (Fig. 3(b)). Each membrane compressor is

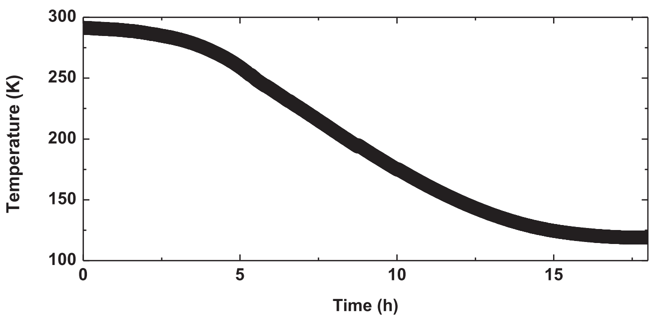

Fig. 5. Temperature versus time taken during an overnight cool down of a 10 T superconducting magnet using the cold Helium closed-cycle system. During the morning, Helium was transferred with the same evaporation rate as found when using liquid nitrogen in the cryostat.

connected to a valve ((3) in Fig. 3b) that opens when the compressor stops to remove the overpressure at the compressor's outlet and equilibrate pressures at inlet and the outlet. Otherwise, the pistons moving the membranes would not be able to restart again. There is a one-way valve connected to the entry of the pressurized container (2) in Fig. 3b. In usual operation, the valves (3) are thus connected from the compressor exits to air. But for our application, we have to connect the valves to the low pressure entry. This allows for leak tight operation of the system without air.

At the exit of the pressurized container there is a pressure guage, a small filter and the switch that is connected to one of the pressure guages. The filter serves to eliminate moisture and particles that are usually present in pressured containers of commercial compressors. All tubes need to be replaced by clean PVC pipings with fittings that can hold enough pressure. The compressor is activated by a pressure sensor installed in the container, that starts the motor at 1.5 bars and stops it at 4 bar. At the exit of the container, a flow regulator allows to establish the needed flow of gas through the circuit. A small filtering unit serves to remove impurities from the gas. A valve opening at 6 bar provides needed safety in case of overpressure.

2.3. Sliding seal assembly and inner vacuum chamber for rapid sample exchange

Often, a refrigerator with diameter well above 100 mm has to be inserted into liquid Helium (see e.g. [19]). This can be made by a system that allows precooling the assembly using Helium gas. To this end, the cryostat needs a cylindrical enclosure with a Helium recovery port and a sliding seal that blocks the vertical motion of the cryostat, particularly if an overpressure appears in the cryostat. At the same time, the system should be leak tight to avoid loss of Helium gas. A solution adopted by several companies is to use a radial shaft spring seal[20] comprised by a plastic ring that is compressed using a spring. The shaft seal, however, is designed for operation in motors, where the leak-tightness is provided by a fluid in which the shaft turns. When using it in a sliding seal assembly there is no lubricant and it often leaks.

Other solutions include the use of bellows and a seal assembly[21,22]. These allow for lateral and rocking motions appearing during transport of a large cryostat assembly but occupy considerably more space, hampering the usual operation when the cryostat is cold.

Here we propose a simple alternative consisting of substituting the shaft spring by an O-Ring assembly comprised of a O-ring clamped using a stainless steel ring. The ring includes an assembly that allows fixing the position of the insert when a overpressure is built up in the Dewar or when the Dewar is pumped.

Furthermore, we propose a new design for the vacuum can of the cryostat that reduces the liquid Helium evaporation rate. The bottom part of a dilution refrigerator contains the experiment and is located inside a vacuum can, often termed the inner vacuum chamber, as opposed to the 'outer' vacuum chamber of the containing Dewar. The inner vacuum chamber is most often made in stainless steel and is partly immersed into liquid Helium. The vacuum chamber connects the upper flange of the chamber to the liquid Helium bath. The upper flange needs usually to be as cold as possible, to facilitate condensation of cryogens that arrive from room temperature through tubing soldered to the upper flange. This is often important, because the liquid Helium level can be well below the said upper flange (the liquid Helium is pumped through a vacuum insulated capillary inside the inner vacuum chamber). To lower the temperature of the upper flange, the solution is often to simply weld a copper tube to the outside of the stainless steel inner vacuum chamber at a few points. The resulting system has a considerable weight, which increases the need of Helium to cool down.

To reduce the weight of the inner vacuum chamber, we have built a chamber out of Aluminum alloy. The chamber comprises an indium seal assembly on the top to connect the chamber to the dilution insert. The chamber also comprises an indium seal on its bottom. The weight of the chamber is reduced by a factor of three with respect to chamber consisting of copper and stainless steel. The enthalpy between room temperature and low temperatures is similar for stainless steel and Aluminum alloys (we have used 6082 and 2030 alloys, both with similar performances). Thus, the amount of heat to be removed for cooling is a factor of three smaller in the case of Aluminum inner vacuum chambers. On the other hand, the thermal conductivity of Aluminum alloy is most often at least an order of magnitude larger than the thermal conductivity of stainless steel, which allows for effective cooling of the upper flange. Using these tubes we achieved improved performances in our cryostats, both by being able to cool faster and by having less heat load into the cold parts inside the inner vacuum chamber when the liquid Helium level is far below the upper flange.

2.4. Acknowledgments

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3. Design files

3.1. Design files summary

Design filename	File type	Open source license	Location of the file
Design file 1	Figure	CERN OHL	Available with the article, see Fig. 1
DesignFile HeatExchanger.pdf and.stl	pdf and stl of CAD plots	CERN OHL	https://osf.io/e6k7r/
DesignFile ConnectorRoom Temperature. pdf and.stl	pdf and stl of CAD plots	CERN OHL	https://osf.io/e6k7r/
DesignFile Sliding Seal1.pdf and.stl	pdf and stl of CAD plots	CERN OHL	https://osf.io/e6k7r/
DesignFile InnerVacuum Chamber1.pdf and.stl	pdf and stl of CAD plots	CERN OHL	https://osf.io/e6k7r/
DesignFileInner Vacuum Chamber2.pdf and.stl	pdf and stl of CAD plots	CERN OHL	https://osf.io/e6k7r/
DesignFileInner Vacuum Chamber3.pdf	pdf of CAD plots	CERN OHL	https://osf.io/e6k7r/
DesignFile Pump.pdf	Pdf with photos showing how to clean the pump and improve its leak tightness	CERN OHL	https://osf.io/e6k7r/

Design file 1: Connecting scheme for the manual pump.

DesignFileHeatExchanger: Design files of the heat exchanger. It includes a design file for connectors for tubes of different diameters.

DesignFileConnectorRoomTemperature: Design files of the connector for the sliding seal that replaces the shaft spring ring.

DesignFileSlidingSeal1: Design file of the sliding seal assembly.

DesignFileInnerVacuumChamber1 to 3: Three possible realizations of inner vacuum chambers.

DesignFilePump: Photographs indicating how to clean the manual pump.

4. Bill of materials

Designator	Component	Number	Cost per unit currency	Total cost	Source of materials	Material type
1	Manual pump	1	20$	20$	Usual sport or camping market	manual pump with 5 liter volume at each stroke and entry and exit ports
2	O-rings, glue and grease	1	30$	30$	Supplier of cryogenic spares and components	Small viton o-rings, Araldite™(blue) glue, Teflon™and vacuum grease
2	Rubber tubes and fittings	2	5$	10$	Magazine supplier	Tubes for the recovery

Designator	Component	Number	Cost per unit currency	Total cost	Source of materials	Material type
1	Compressor Stayer 70	1	280$	280$	Building market	High pressure machine
2	Electrovalve SMC position normally open	2	50$	100$	Compressed air products	Valves
3	Presostat	1	50$	50$	Compressed air products	Sensor and valves
4	Balloon	1	1000$	1000$	Balloon made of quality rubber	Gas recipient
5	Fittings and pipes	–	70$	70$	–	–

Designator	Component	Number	Cost per unit currency	Total cost	Source of materials	Material type
1	Machining hardware	1	200$	200$	Hardware to machine the shown items. In addition, personnel costs can apply.	–

5. Build instructions

5.1. Manual pump

- Remove grease from the entry and exit ports of the manual pump.
- Open the pump using a strap wrench.
- Remove all grease from the inside of the pump.
- Apply vacuum grease to all moving parts of the pump.
- Add Teflon™ and an O-ring to the large diameter screwcap of the pump.
- Use Araldite™ to make the pressure reading leak tight, putting epoxy on the juncture between the manometer and the tube.
- Use Araldite™ to glue connectors for flexible rubber tubes at the entry and exit ports.

5.2. Closed cycle circuit of cold Helium

- Substitute PVC tubes of the compressor with copper pipes, 6 mm diameter. Use conical copper fittings and make the required adaptations between tube diameters. One might also use sylicone tubing with high pressure conical fittings.
- Replace the pressostat of the compressor with a type Danfoss Kp35 060 pressostat or similar, a safety vale (8 bar) and a filter with flow controller and valve (a usual pneumatic filter, such as SMC AW20).
- Replace the electrovalves of the compressor. Often, valves from a commercial and cheap air compressor are leaking. Replace these with electrovalves that do not leak. Use SMC normally open valves. Be careful not to substitute the one-way valve between the compressors and the pressurized container.
- Remove all water from the pressurized container and block the draining system by gluing a screw to it.
- When replacing items such as valves and tubes, remember to seal with Araldite™or similar all threads where the valves are screwed. Verify all the compressor for entry and exit ports, remove eventual screwcaps and seal these using Araldite™ or similar.
- Sometimes there are two separate entry ports, one for each membrane compressor. In that case, connect the two entry ports of the compressor to a single line using a T.

5.3. Sliding seal assembly and inner vacuum chamber

- Remove the shaft spring seal and make the stainless steel pieces that hold the O-Ring as shown in the design files.
- Obtain an O-ring that exactly fits to the outer diameter of the sliding seal.
- For the Aluminum inner vacuum chamber, make the pieces as shown in the design files. Note that it is better to use an Indium seal at the bottom. Otherwise, it is difficult to maintain the same dimensions as the original stainless steel vacuum chamber.
- Seal the bottom of the inner vacuum chamber with an Indium seal. Mount the chamber to the cryostat through the top Indium seal.

6. Operation instructions

- *Precautions*
- Usual precautions when using high pressure equipment. Take into account local high pressure safety regulations, usually described in the manual of the commercial compressor. In particular, safety valves should be set at the specified values.
- The manual pump does not require specific precautions.
- When inserting the cryostat into liquid Helium always hold the cryostat by a thread so that it is not pushed out in presence of overpressure at the Helium recovery lines.
- *Operation of the pump*
- Connect the entry of the manual pump to the recovery line and the exit to the transport Dewar.
- When pumping, take care not to build an overpressure in the transport Dewar. This is very unlikely, because all transport Dewars have adequate safety valves and because the pump must be operated many times to create a significant overpressure. But it should nevertheless be noted.

- *Operation of the closed cycle circuit*
- First of all, the operator should make sure that the pressurized container ((2) in Fig. 2) is filled with 4 bar of Helium. If this is not the case, the entry of the compressor should be connected to the recovery line and the pressurized container filled with Helium by opening valve (7) in Fig. 2 (with valve (3) closed).
- The transfer siphon (5) in Fig. 2 should be inserted into the heat exchanger and the cryostat.
- The siphon should be inserted down to the bottom end of the cryostat, so that the cold Helium flows from the bottom to the top of the cryostat.
- The cryostat's exit of Helium should be connected to the balloon (8) in Fig. 2.
- It is very important to make now sure that valve (7) is closed, so that no Helium is pumped from the recovery line.
- Valve (3) in Fig. 2 should then be opened and the compressor switched on.
- The balloon (8) in Fig. 2 will start filling and the pressure in the container (2) in Fig. 2 will decrease.
- Adjust valve (3) in Fig. 2 and make sure that the compressor starts when the container's pressure decreases below approximately 1.5 bar and stops when it reaches 4 bar.
- Circulate Helium during at least 15 min and make sure that there are no leaks, particularly in the connection between the siphon and the heat exchanger, as well as between the siphon and the cryostat.
- When it is clear that there are no leaks, insert slowly the heat exchanger into a liquid nitrogen dewar.
- When the heat exchanger is cold, make sure that there is a significant evaporation of liquid nitrogen. Please remind that this method is in no way reducing the consumption of liquid nitrogen. One needs to evaporate as much liquid nitrogen as when it is directly inserted in the cryostat. Therefore, the liquid nitrogen container should be well vented and there will be a significant amount of nitrogen gas flowing out of it.
- You might readjust valve (3) in Fig. 2 to find the best pressures for compressor start and stop in your system. Seek for maximizing the flow of Helium gas and of the evaporation of liquid nitrogen.
- Monitor the temperature in the cryostat (6) making sure that it drops.
- The system can be left overnight as such.
- Blockages as described in the following are rare and can be avoided by allowing the system running for a while at room temperature and carefully checking for leaks. In the unlikely event that this happens, it is usually identified by absence of liquid nitrogen evaporation in the liquid nitrogen container. If this occurs, close valve (3) in Fig. 2 and remove at the same time the heat exchanger (4) from the liquid nitrogen container and the siphon (5) from cryostat. Make sure that you immediately put a cap in the cryostat's entry for the siphon. Heat using a heat gun, always leaving the entry and exit ports vented. When it is warm, remove the siphon and use pressurized air or nitrogen to remove all water from the heat exchanger. Connect again the heat exchanger and the siphon ((5) in Fig. 2) to the heat exchanger and open valve ((3) in Fig. 2). Allow for some gas going out of the exist of the siphon and enter the siphon into the cryostat. It might happen that some Helium gas is lost during this procedure. Then, the pressure at the pressurized container might never reach high values. Then, open valve (7) in Fig. 2 to refill the system with Helium gas.
- *Operation of the sliding seal assembly*
- Disconnect the system from the Helium recovery line. Remove the cap and introduce slowly the insert into the cryostat until the bottom of the sliding seal reaches the O-ring seal.
- Close the O-ring seal and connect again to the Helium recovery line. Wait for about 15 min and check for leaks at the O-ring.
- Lower the insert in steps of about 10 cm each. Wait for at least 15 min at each step.
- If a leak appears, it might be due to frozen water. Then, disconnect the system from the Helium recovery line to avoid overpressure and heat the top of the cryostat until all moisture is gone. If needed, tight the O-ring seal further using the screws in the upper flange of the O-ring seal assembly.
- When the bottom of the insert touches liquid Helium, usually a large boil-off starts. Be careful that the seal does not leak at this point and that the insert is not pushed upwards.

7. Validation and characterization

- We have observed that we can have transfer rates as high as 100 liters an hour operating a few times the manual pump. When the Helium transfer Dewar is nearly empty, the pump must be operated a few more times, as the volume needed to fill is much larger. We have used the manual pump in 50 liter and 100 liter transport Dewars. We do not expect major changes when using it in a 200 liter transport Dewar.
- In Fig. 5 we show a typical cool down procedure to liquid nitrogen using the closed-cycle system. A bottle of about 60 liters of liquid nitrogen was emptied during the night.
- We have observed a decrease of the time needed to cool a cryostat by a factor of three when using the Aluminum inner vacuum chamber. In addition, the time needed to condense the mixture is reduced by a factor of two with respect to the use of stainless steel inner vacuum chambers.

Declaration of interest

None.

Human and animal rights

Does not apply.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, athttps://doi.org/10.1016/j.ohx.2019.e00058.

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